JP WEBSITE Machine Translation of JP 2001-1898-20877

[Claim(s)]
[Claim 1]A following general formula [I] A bathophenanthroline compound expressed.
[Formula 1]
- 歌武 (1):

(However, said general formula) [I] Set and R^1 and R^2 , it is mutually the same, or it is a different basis, and is a hydrocarbon group of the saturation which is not replaced [the hydrocarbon group of a straight chain, branching, annular saturation, or an unsaturation, substitution, or], or an unsaturation, and at least one carbon number of R^1 and R^2 is two or more.

[Claim 2]A following general formula [III] A bathophenanthroline compound expressed.

[Formula 2]

— #st [III] :

(However, said general formula) [II] Setting, Ar^1 and Ar^2 are aryl groups which are the same or are mutually different and which is not replaced [substitution or].

[Claim 3]A bathophenanthroline compound indicated to claim 1 or 2. [available as organic materials used for an organic electrolysis light emitting device, for example, a carrier transport material, 1 [Claim 4]A following general formula [III] A lithium compound expressed and a following general formula [IV] By carrying out the nucleophilic substitution of the bathophenanthroline expressed, it is a following general formula. [IV] A manufacturing method of a bathophenanthroline compound which obtains a bathophenanthroline compound expressed.

[Formula 4]

一般式〔〕:



(However, said general formula) [I] Set and R^1 and R^2 , it is mutually the same, or it is a different basis, and is a hydrocarbon group of saturation which is not replaced [a hydrocarbon group of a straight chain, branching, annular saturation, or an unsaturation, substitution, or], or an unsaturation, and at least one carbon number of R^1 and R^2 is two or more.

[Claim 5]A following general formula [V] A lithium compound expressed and a following general formula [IV] By carrying out the nucleophilic substitution of the bathophenanthroline expressed, it is a following general formula. [II] A manufacturing method of a bathophenanthroline compound which obtains a bathophenanthroline compound expressed.

A general formula [V]: Ar³-Li or Ar⁴-Li (however, said general formula) [V] Setting, Ar³ and Ar⁴ are aryl groups which are the same or are mutually different and which is not replaced [substitution or].

[Formula 5]

[Formula 6] 一般式 (II) ;

(However, said general formula) [II] Setting, Ar^1 and Ar^2 are aryl groups which are the same or are mutually different and which is not replaced [substitution or].

[Claim 6]A manufacturing method of a bathophenanthroline compound which generates carbanion with said lithium compound in a solution, to which this carbanion and said bathophenanthroline are made to react in said nucleophilic substitution and which was indicated to claim 4 or 5.

[Translation done.]

DETAILED DESCRIPTION

[Detailed Description of the Invention]

[Field of the Invention] This invention is organic electroluminescence devices (for example, it is a flatsurface type display of spontaneous light, and). It is related with a suitable bathophenanthroline [to use an organic thin film for display devices, such as an organic electroluminescence color display used for an electroluminescence layer, or the suitable organic electroluminescence devices for a luminescent device especially] compound, and a manufacturing method for the same. [0002]

[Description of the Prior Art]In recent years, the importance of an interface to human being and a machine is increasing by making multimedia-oriented goods into the start. In order for human being to do mechanical control efficiently more comfortably, research is done about various display devices which there is no error, and need to take [an instant and] out the information from the machinery operated in sufficient quantity briefly, therefore make a display the start.

[0003]The actual condition is that the demand to the miniaturization of a display device and slimming down is also increasing with a mechanical miniaturization every day.

[0004] For example, the miniaturization of the laptop type information management system which are display device integral types, such as a note type personal computer and a note type word processor, is astonishing, and the technical innovation about the liquid crystal display which is the display device also has a wonderful thing in connection with it.

[0005] The liquid crystal display is used as an interface of various products today.

Not to mention the laptop type information management system, it is mostly used for the product used our every day by making small television, a clock, and a calculator into the start.

[0006]These liquid crystal displays have been studied as a center of a display device as an interface of human being and machinery from small size to a mass display device taking advantage of the feature that liquid crystals are a low voltage drive and low power consumption.

[0007]However, since this liquid crystal display is not self-luminescence, it needs a back light, since big electric power is needed for this back light drive rather than driving a liquid crystal, as a result, by a built-in storage battery, a hour of use becomes short and it has the restriction on use.

[0008] Since the angle of visibility of a liquid crystal display is narrow, it is a problem that it is not suitable for large-sized display devices, such as a large sized display, either.

[0009]It is thought that a liquid crystal display is a problem in which it is also big that contrast changes with angles into an angle of visibility since it is the method of presentation by the oriented state of a liquid crystal element.

[0010]If it thinks from a drive system, although sufficient speed of response to treat an animation is shown, in order to use a TFT (thin film transistor) drive circuit, enlargement of screen size is difficult for the active matrix which is one of the drive systems by a picture element defect, and it is disadvantageous also in terms of a cost cut.

[0011]In a liquid crystal display, although the passive matrix which is another drive system is low cost and enlargement of screen size is comparatively easy for it, there is a problem that it does not have sufficient speed of response to treat an animation.

[0012]On the other hand, as for the self-luminescence display device, a plasma display device, inorganic electroluminescence devices, organic electroluminescence devices, etc. are studied.

[0013] Although a plasma display device is what used the plasma emission in the inside of low pressure gas for the display and it is suitable for enlargement and large scale-ization, it has the problem in the field of slimming down and cost. The AC bias of high tension is needed for a drive, and it is not suitable for a nortable device.

[0014]Inorganic electroluminescence devices are AC bias drives like a plasma display device, although the green emission display etc. were commercialized.

Hundreds of v is required for a drive, and practicality is missing.

[0015]However, although a success is carried out to luminescence of the three primary colors of R (red) required for a color display display, G (green), and B (blue) by technical development, it seems that control of the luminous wavelength by a molecular design etc., etc. is difficult because of an inorganic material, and full-color-izing is difficult.

[0016] on the other hand, the luminous phenomenon by career pouring to the anthracene single crystal in which the electroluminescence phenomenon by an organic compound generates fluorescence in the first half of the 1960s strongly is discovered — since then, a long period, although it has inquired, Since it was low-intensity and monochrome and was moreover a single crystal, it was carried out as fundamental study called career pouring to organic materials.

[0017]However, Tang and others of Eastman Kodak has announced the organic thin film electroluminescence devices of the laminated structure which has an amorphous luminous layer in which a low voltage drive and bright luminescence are possible, and them in 1987 in every direction since then, Research and development of luminescence of the three primary colors of R, G, and B, stability, a luminosity rise, a laminated structure, a manufacturing method, etc. are done briskly. [0018]Although it is the feature of organic materials, the application study to the color display of the organic electroluminescence display device which various new materials are invented by the molecular design etc. and has the feature which was excellent in a DC voltage drive, a thin shape, self-luminescence, etc. is also beginning to be performed briskly.

[0019]Organic electroluminescence devices (an organic EL device may be called hereafter.) are 1 micrometer or less of thickness.

It has the feature ideal as a spontaneous light [, such as transforming electrical energy into light energy and emitting light to surface state,] type display device by pouring in current.

[0020]Drawing 7 shows an example of the conventional organic EL device 10. This organic EL device 10 forms the ITO (Indium tin oxide) transparent electrode 5, the hole transporting bed 4, the luminous layer 3, the electron transport layer 2, and the negative pole (for example, aluminum electrode) 1 one by one with a vacuum deposition method on the transparent substrate (for example, glass substrate) 6. [0021]And by impressing the direct current voltage 7 selectively between the transparent electrode 5 and the negative pole 1 which are the anode, The electron in which the hole as a career poured in from the transparent electrode 5 was poured in from the negative pole 1 through the hole transporting bed 4 moves through the electron transport layer 2, the recombination of an electronic-hole arises, the luminescence 8 of a prescribed wavelength arises from here, and it can observe from the transparent substrate 6 side.

[0022]Photogene, such as antiracene, naphthalene, phenanthrene, pyrene, a chrysene, perylene, butadiene, a coumarin, an acridine, and a stilbene, may be used for the luminous layer 3, for example. The electron transport layer 2 can be made to contain this.

[0023]Drawing 8 shows another conventional example.

Omit the luminous layer 3, the electron transport layer 2 is made to contain the photogene like the above, and the organic EL device 20 constituted so that the luminescence 18 of a prescribed wavelength might arise from the interface of the electron transport layer 2 and the hole transporting bed 4 is shown.

[0024]Drawing 9 shows the example of the above-mentioned organic EL device. Namely, although the layered product of each organic layer (the hole transporting bed 4, the luminous layer 3, or the electron transport layer 2) is allotted between the negative pole 1 and the anode 5, These electrodes are made to intersect matrix form, it provides in stripe shape, and a signal level is impressed to a time series by the luminance-signal circuit 34 and the control circuit 35 with a built-in shift register, and it constitutes so that light may be made to emit in many intersecting positions (pixel), respectively.

[0025]Therefore, of course, it becomes usable also as picture reproducer as a display by such composition. The above-mentioned stripe pattern is arranged for every color of R, G, and B, and it can be full color or can constitute as an object for multicolor.

[0026]In the display device using such an organic EL device which consists of two or more pixels,

generally, the organic thin film layers 2, 3, and 4 which emit light are inserted between the transparent electrode 5 and the metal electrode 1, and emit light to the transparent electrode 5 side. 100271

[Problem(s) to be Solved by the Invention]However, concern about an organic luminescent material and a suitable carrier transport material for an organic luminescent material is increasing as a component of the above organic EL devices, and the advantage of these organic materials is in the place which can be optical and can control electric nature to some extent by a molecular design. Therefore, luminescence can be efficiently obtained by combining the organic luminescent material which has predetermined luminescence, and a carrier transport material suitable for it. By this, it becomes realizable [the full color organic EL device which created all three-primary-colors luminescence of R, G and B with each luminescent material].

[0028]As a feature of such an organic EL device, an electron hole (hole) transporting bed may have the element structure which serves as a light emitting device. In such element structures, the carrier transport laver which conveys an electron efficiently and blocks a hole is required.

[0029]However, the organic materials which fill such a demand, and the method of manufacturing this efficiently are not yet found out.

[0030]Then, the purpose of this invention is to provide new organic materials suitable as a carrier transport material which conveys an electron efficiently and blocks a hole, and the method of manufacturing this efficiently.

[0031]

[Means for Solving the Problem]That is, this invention is a following general formula. [I]or[II] Start a bathophenanthroline compound expressed.

[Formula 7]

(However, said general formula) [I] Set and R^1 and R^2 , it is mutually the same, or it is a different basis, and is a hydrocarbon group of the saturation which is not replaced [the hydrocarbon group of a straight chain, branching, annular saturation, or an unsaturation, substitution, or], or an unsaturation, and at least one carbon number of R^1 and R^2 is two or more.

[Formula 8] 一般式 (II) :

(However, said general formula) [II] Setting, Ar^1 and Ar^2 are aryl groups which are the same or are mutually different and which is not replaced [substitution or].

[0032]A bathophenanthroline compound of this invention is available as a carrier transport material of various organic electroluminescence devices from carrier transport nature being controllable depending

on a substituent introduced into the intramolecular. Such materials have a high glass transition point and the melting point, electrically, it is thermal or chemically stable, and also are sublimability, and have an advantage which can form uniform amorphous films easily by a vacuum deposition process.

[0033]As for R^1 and R^2 , Ar^1 , and Ar^2 , in a bathophenanthroline compound of this invention, it is preferred that it is the same. Here, an "aryl group" means heterocyclic aromatic groups, such as carbocyclic aromatic groups, for example, a furil group, such as a phenyl group, a naphthyl group, and an anthryl group, a thienyl group, and a pyridyl group, for example.

[0034]This invention is a following general formula. [III]or[V] A lithium compound expressed and a following general formula [IV] By carrying out the nucleophilic substitution of the bathophenanthroline expressed, it is said general formula. [I]or[II] Also provide a manufacturing method of a bathophenanthroline compound which obtains a bathophenanthroline compound expressed.

General formula[III]: R³-Li or R⁴-Li (however, said general formula) [III] Set and R³ and R⁴, it is mutually the same, or it is a different basis, and is a hydrocarbon group of saturation which is not replaced [a hydrocarbon group of a straight chain, branching, annular saturation, or an unsaturation, substitution, or], or an unsaturation, and at least one carbon number of R³ and R⁴ is two or more. General formula[V]: Ar³-Li or Ar⁴-Li (however, said general formula) [V] Setting, Ar³ and Ar⁴ are aryl groups which are the same or are mutually different and which is not replaced [substitution or].

[Formula 9]

—882 [IV]:

[0035] With the manufacturing method of this invention, the bathophenanthroline compound of this invention can be manufactured efficiently. In this case, in nucleophilic substitution, it is good to generate carbanion with said lithium compound in a solution, and to make this carbanion and said bathophenanthroline react.

[0036]A general formula among bathophenanthroline compounds of this invention [I] In \mathbb{R}^1 and \mathbb{R}^2 of a compound which are expressed as an example of a hydrocarbon group of branching, annular saturation, or an unsaturation, For example, an ethyl group, a butyl group, n-propyl group, an isopropyl group, n-butyl group, A sec-butyl group, a tert-butyl group, n-pentyl group, an isopentyl group, A neopentyl group, a tert-pentyl group, a cyclopentylic group, n-hexyl group, 2-ethylbutyl group, a 3,3-dimethylbutyl group, a cyclohexyl group, Although n-heptyl group, a cyclohexylmethyl group, n-octyl group, a tert-octyl group, a 2-ethylbutyl group, n-hexyl group, n-decyl group, n-hexyl gr

[0037]As an example of a hydrocarbon group of saturation which is not replaced [substitution of \mathbb{R}^1 and \mathbb{R}^2 , or], or an unsaturation, For example, benzyl, phenethyl group, alpha-methylbenzyl group, group, alpha, and alpha-dimethylbenzyl group, 1-naphthyl methyl group, 2-naphthyl methyl group, a furfuryl group, 2-methylbenzyl group, 3-methylbenzyl group, 3-methylbenzyl group, 4-methylbenzyl group, 4-thylbenzyl, at can do [mentioning a hydrocarbon group of saturation such as 4-isopropylbenzyl, a 4-tert-butylbenzyl group, 4-n-hexylbenzyl, 4-nonylbenzyl, and a 3,4-dimethylbenzyl group, or an unsaturation, or], it is not limited to these.

[0038]General formula[II] In Ar¹ and Ar² of a compound which are expressed, As an example of an aryl group which is not replaced [substitution or], for example A phenyl group, 1-naphthyl group, 2-anthryl group, 9-anthryl group, 2-fluorenyl group, 4-quinolyl group, 4-pyridyl group, 3-pyridinyl group, 2-

pyridinyl group, 3-furil group, 2-furil group, 3-thienyl group, 2-oxazolyl group, 2-thiazolyl group, 2-benzo oxazole group, 2-benzothiazolyl group, 2-benzo imidazolyl group, 4-methylphenyl group, 3-methylphenyl group, 2, 3-dimethylphenyl group, 2, 4-dimethylphenyl group, 2, 5-dimethylphenyl group, 2, 4-dimethylphenyl group, 3, 5-dimethylphenyl group, 3, 5-dimethylphenyl group, 3, 5-dimethylphenyl group, 3, 6-trimethyl phenyl group, 2 and 3, 5-trimethyl phenyl group, 2, 3, 6-trimethyl phenyl group, 3 and 4, 5-trimethyl phenyl group, 4-ethyl phenyl group, 3-ethyl phenyl group, 2-ethyl phenyl group, 3, 2,3-diethyl phenyl group, 4-diethyl phenyl group, 3,5-diethyl phenyl group, 2, 3,5-triethyl phenyl group, 3,5-diethyl phenyl group, 3,5-diethyl phenyl group, 3,5-triethyl phenyl group, 3,5-triethyl phenyl group, 3,6-triethyl group, 3,5-triethyl group, 3,6-triethyl group, 3,6-triethyl phenyl group, 3,6-triethyl group,

[0039]As a bathophenanthroline compound of this invention, although the following compounds (illustration compounds 1-178) can be mentioned, for example, this invention is not limited to these (however, as for Me, a methyl group and Et express an ethyl group, Pr expresses a propyl group, and Bu

例示化合物 6

expresses a butyl group.).

例示化合物 5

例示化合物 4 [0041] [Formula 11]

[Formula 17]

[0048] [Formula 18]

[0049] [Formula 19]

例示化合物60

例示化合物59

[0050] [Formula 20]

Bt

例示化合物69

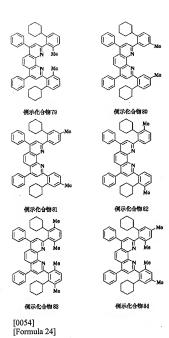
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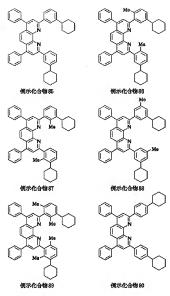


例示化合物71

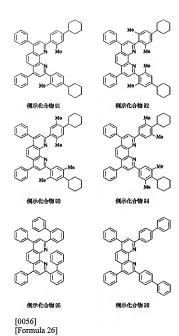
例示化合物72

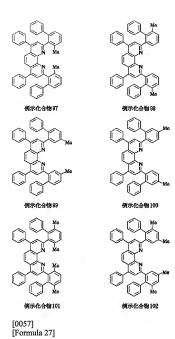
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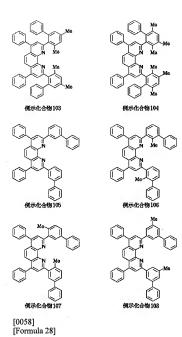


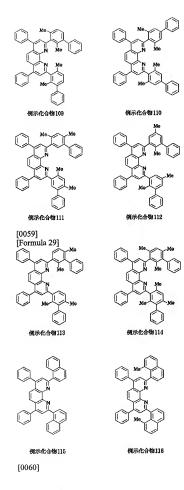


[0055] [Formula 25]









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例示化合物117

例示化合物118

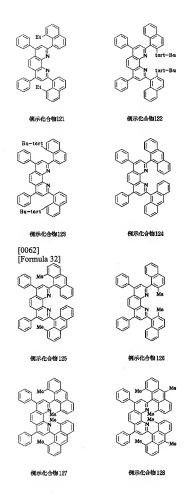


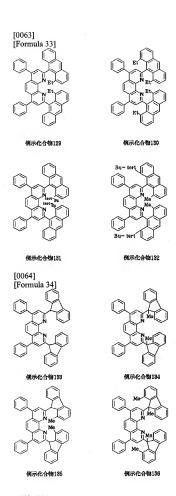


例示化合物119

例示化合物120

[0061] [Formula 31]





[0065] [Formula 35]

例示化合物 187

例示化合物 138

例示化合物189

例示化合物 140

[0066] [Formula 36] CH2 Me

例示化合物141

例示化合物142





例示化合物148

例示化合物144

[0067]

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例示化合物 159 TECHNICAL FIELD 例示化合物 160

3/6/2009

[Field of the Invention] This invention is organic electroluminescence devices (for example, it is a flatsurface type display of spontaneous light, and). It is related with a suitable bathophenanthroline [to use an organic thin film for display devices, such as an organic electroluminescence color display used for an electroluminescence layer, or the suitable organic electroluminescence devices for a luminescent device especially compound, and a manufacturing method for the same.

[Translation done.]

PRIOR ART

[Description of the Prior Art]In recent years, the importance of an interface to human being and a machine is increasing by making multimedia-oriented goods into the start. In order for human being to do mechanical control efficiently more comfortably, research is done about various display devices which there is no error, and need to take [an instant and] out the information from the machinery operated in sufficient quantity briefly, therefore make a display the start.

[0003]The actual condition is that the demand to the miniaturization of a display device and slimming down is also increasing with a mechanical miniaturization every day.

[0004]For example, the miniaturization of the laptop type information management system which are display device integral types, such as a note type personal computer and a note type word processor, is astonishing, and the technical innovation about the liquid crystal display which is the display device also has a wonderful thing in connection with it.

[0005] The liquid crystal display is used as an interface of various products today.

Not to mention the laptop type information management system, it is mostly used for the product used our every day by making small television, a clock, and a calculator into the start.

[0006]These liquid crystal displays have been studied as a center of a display device as an interface of human being and machinery from small size to a mass display device taking advantage of the feature that liquid crystals are a low voltage drive and low power consumption.

[0007]However, since this liquid crystal display is not self-luminescence, it needs a back light, since big electric power is needed for this back light drive rather than driving a liquid crystal, as a result, by a built-in storage battery, a hour of use becomes short and it has the restriction on use.

[0008]Since the angle of visibility of a liquid crystal display is narrow, it is a problem that it is not suitable for large-sized display devices, such as a large sized display, either.

[0009]It is thought that a liquid crystal display is a problem in which it is also big that contrast changes with angles into an angle of visibility since it is the method of presentation by the oriented state of a liquid crystal element.

[0010]If it thinks from a drive system, although sufficient speed of response to treat an animation is shown, in order to use a TFT (thin film transistor) drive circuit, enlargement of screen size is difficult for the active matrix which is one of the drive systems by a picture element defect, and it is disadvantageous also in terms of a cost cut.

[0011]In a liquid crystal display, although the passive matrix which is another drive system is low cost and enlargement of screen size is comparatively easy for it, there is a problem that it does not have sufficient speed of response to treat an animation.

[0012]On the other hand, as for the self-luminescence display device, a plasma display device, inorganic electroluminescence devices, organic electroluminescence devices, etc. are studied.

[0013]Although a plasma display device is what used the plasma emission in the inside of low pressure gas for the display and it is suitable for enlargement and large scale-ization, it has the problem in the field of slimming down and cost. The AC bias of high tension is needed for a drive, and it is not suitable for a portable device.

[0014]Inorganic electroluminescence devices are AC bias drives like a plasma display device, although the green emission display etc. were commercialized.

Hundreds of v is required for a drive, and practicality is missing.

luminescence, etc. is also beginning to be performed briskly.

[0015]However, although a success is carried out to luminescence of the three primary colors of R (red) required for a color display display, G (green), and B (blue) by technical development, it seems that control of the luminous wavelength by a molecular design etc., etc. is difficult because of an inorganic material, and full-color-izing is difficult.

[0016]on the other hand, the luminous phenomenon by career pouring to the anthracene single crystal in which the electroluminescence phenomenon by an organic compound generates fluorescence in the first half of the 1960s strongly is discovered -- since then, a long period, although it has inquired, Since it was low-intensity and monochrome and was moreover a single crystal, it was carried out as fundamental study called career pouring to organic materials.

[0017]However, Tang and others of Eastman Kodak has announced the organic thin film electroluminescence devices of the laminated structure which has an amorphous luminous layer in which a low voltage drive and bright luminescence are possible, and them in 1987 in every direction since then, Research and development of luminescence of the three primary colors of R, G, and B, stability, a luminosity rise, a laminated structure, a manufacturing method, etc. are done briskly. [0018] Although it is the feature of organic materials, the application study to the color display of the organic electroluminescence display device which various new materials are invented by the molecular design etc. and has the feature which was excellent in a DC voltage drive, a thin shape, self-

[0019]Organic electroluminescence devices (an organic EL device may be called hereafter.) are I micrometer or less of thickness.

It has the feature ideal as a spontaneous light [, such as transforming electrical energy into light energy and emitting light to surface state,] type display device by pouring in current.

[0020] Drawing 7 shows an example of the conventional organic EL device 10. This organic EL device 10 forms the ITO (Indium tin oxide) transparent electrode 5, the hole transporting bed 4, the luminous layer 3, the electron transport layer 2, and the negative pole (for example, aluminum electrode) 1 one by one with a vacuum deposition method on the transparent substrate (for example, glass substrate) 6. [0021] And by impressing the direct current voltage 7 selectively between the transparent electrode 5 and the negative pole 1 which are the anode, The electron in which the hole as a career poured in from the transparent electrode 5 was poured in from the negative pole 1 through the hole transporting bed 4 moves through the electron transport layer 2, the recombination of an electronic-hole arises, the luminescence 8 of a prescribed wavelength arises from here, and it can observe from the transparent substrate 6 side.

[0022]Photogene, such as anthracene, naphthalene, phenanthrene, pyrene, a chrysene, perylene, butadiene, a coumarin, an acridine, and a stilbene, may be used for the luminous layer 3, for example. The electron transport layer 2 can be made to contain this.

[0023]Drawing 8 shows another conventional example.

Omit the luminous layer 3, the electron transport layer 2 is made to contain the photogene like the above, and the organic EL device 20 constituted so that the luminescence 18 of a prescribed wavelength might arise from the interface of the electron transport layer 2 and the hole transporting bed 4 is shown.

[0024] <u>Drawing 9</u> shows the example of the above-mentioned organic EL device. Namely, although the layered product of each organic layer (the hole transporting bed 4, the luminous layer 3, or the electron transport layer 2) is allotted between the negative pole 1 and the anode 5, These electrodes are made to intersect matrix form, it provides in stripe shape, and a signal level is impressed to a time series by the luminance-signal circuit 34 and the control circuit 35 with a built-in shift register, and it constitutes so that light may be made to emit in many intersecting positions (pixel), respectively.

[0025]Therefore, of course, it becomes usable also as picture reproducer as a display by such composition. The above-mentioned stripe pattern is arranged for every color of R, G, and B, and it can be full color or can constitute as an object for multicolor.

[0026]In the display device using such an organic EL device which consists of two or more pixels, generally, the organic thin film layers 2, 3, and 4 which emit light are inserted between the transparent electrode 5 and the metal electrode 1, and emit light to the transparent electrode 5 side.

[Translation done.]

EFFECT OF THE INVENTION

[Function and Effect of the Invention] The bathophenanthroline compound of this invention is available as a carrier transport material of various organic electroluminescence devices from carrier transport nature being controllable depending on the substituent introduced into the intramolecular. Such materials have a high glass transition point and melting point, it is electric, thermal, or chemically stable, and also are sublimability, and have an advantage which can form uniform amorphous films easily by a vacuum deposition process. The bathophenanthroline compound of this invention can be efficiently manufactured using the nucleophilic substitution which used organic lithium.

TECHNICAL PROBLEM

[Problem(s) to be Solved by the Invention]However, concern about an organic luminescent material and a suitable carrier transport material for an organic luminescent material is increasing as a component of the above organic EL devices, and the advantage of these organic materials is in the place which can be optical and can control electric nature to some extent by a molecular design. Therefore, luminescence can be efficiently obtained by combining the organic luminescent material which has predetermined luminescence, and a carrier transport material suitable for it. By this, it becomes realizable [the full color organic EL device which created all three-primary-colors luminescence of R, G and B with each luminescent material].

[0028]As a feature of such an organic EL device, an electron hole (hole) transporting bed may have the element structure which serves as a light emitting device. In such element structures, the carrier transport layer which conveys an electron efficiently and blocks a hole is required. [0029]However, the organic materials which fill such a demand, and the method of manufacturing this

efficiently are not yet found out.

[0030]Then, the purpose of this invention is to provide new organic materials suitable as a carrier transport material which conveys an electron efficiently and blocks a hole, and the method of manufacturing this efficiently.

[Translation done.]

* NOTICES *

JPO and INPIT are not responsible for any damages caused by the use of this translation.

- 1. This document has been translated by computer. So the translation may not reflect the original precisely.
- 2.**** shows the word which can not be translated.
- 3.In the drawings, any words are not translated.

MEANS

[Means for Solving the Problem]That is, this invention is a following general formula. [I]or[II] Start a bathophenanthroline compound expressed. [Formula 7]

(However, said general formula) [I] Set and R^1 and R^2 , it is mutually the same, or it is a different basis, and is a hydrocarbon group of the saturation which is not replaced [the hydrocarbon group of a straight chain, branching, annular saturation, or an unsaturation, substitution, or], or an unsaturation, and at least one carbon number of R^1 and R^2 is two or more.

[Formula 8] 一般式 (II) :

(However, said general formula) [II] Setting, Ar¹ and Ar² are aryl groups which are the same or are mutually different and which is not replaced [substitution or].

[0032]A bathophenanthroline compound of this invention is available as a carrier transport material of various organic electroluminescence devices from carrier transport nature being controllable depending on a substituent introduced into the intramolecular. Such materials have a high glass transition point and the melting point, electrically, it is thermal or chemically stable, and also are sublimability, and have an advantage which can form uniform amorphous films easily by a vacuum deposition process.

[0033]As for R^1 and R^2 , Ar^1 , and Ar^2 , in a bathophenanthroline compound of this invention, it is preferred that it is the same. Here, an "aryl group" means heterocyclic aromatic groups, such as carbocyclic aromatic groups, for example, a furil group, such as a phenyl group, a naphthyl group, and an anthryl group, a thienyl group, and a pyridyl group, for example.

[0034]This invention is a following general formula. [III]or[V] A lithium compound expressed and a following general formula [IV] By carrying out the nucleophilic substitution of the bathophenanthroline expressed, it is said general formula. [I]or[II] Also provide a manufacturing method of a bathophenanthroline compound which obtains a bathophenanthroline compound expressed. General formula[III]: R³-Li or R⁴-Li (however, said general formula) [III] Set and R³ and R⁴, it is mutually the same, or it is a different basis, and is a hydrocarbon group of saturation which is not replaced [a hydrocarbon group of a straight chain, branching, annular saturation, or an unsaturation, substitution, or 1, or an unsaturation, and at least one carbon number of R³ and R⁴ is two or more.

General formula[V]: Ar³-Li or Ar⁴-Li (however, said general formula) [V] Setting, Ar³ and Ar⁴ are aryl



[0035]With the manufacturing method of this invention, the bathophenanthroline compound of this invention can be manufactured efficiently. In this case, in nucleophilic substitution, it is good to generate carbanion with said lithium compound in a solution, and to make this carbanion and said bathophenanthroline react.

[0036]A general formula among bathophenanthroline compounds of this invention [I] $\ln R^1$ and R^2 of a compound which are expressed as an example of a hydrocarbon group of branching, annular saturation, or an unsaturation, For example, an ethyl group, a butyl group, n-propyl group, an isopropyl group, n-butyl group, A sec-butyl group, a et-butyl group, n-pentyl group, an isopentyl group, A neopentyl group, a et-t-pentyl group, a cyclopentylic group, n-hexyl group, 2-ethylbutyl group, a 3,3-dimethylbutyl group, a cyclohexyl group, Although n-heptyl group, a cyclohexylmethyl group, n-octyl group, a tert-octyl group, a 2-ethylhexyl group, n-nonyl group, n-decyl group, n-dedecyl, n-tetradecyl group, n-hexadecyl group, etc. can be mentioned, it is not limited to these.

[0037]As an example of a hydrocarbon group of saturation which is not replaced [substitution of \mathbb{R}^1 and \mathbb{R}^2 , or], or an unsaturation, For example, benzyl, phenethyl group, alpha-methylbenzyl group, group, alpha, and alpha-dimethylbenzyl group, 1-naphthyl methyl group, 2-naphthyl methyl group, 3-methylbenzyl group, 3-methylbenzyl group, 3-methylbenzyl group, 3-methylbenzyl group, 3-methylbenzyl group, 4-methylbenzyl group, 4-thylbenzyl, at 4-tert-butylbenzyl group, 4-n-bexylbenzyl, 4-nonylbenzyl, and a 3,4-dimethylbenzyl group, or an unsaturation, or], it is not limited to these.

[0038]General formula[II] In Ar¹ and Ar² of a compound which are expressed, As an example of an aryl group which is not replaced [substitution or], for example A phenyl group, 1-naphthyl group, 2-anthryl group, 9-anthryl group, 2-fluorenyl group, 4-quinolyl group, 4-pyridyl group, 3-pyridinyl group, 2pyridinyl group, 3-furil group, 2-furil group, 3-thienyl group, 2-oxazolyl group, 2-thiazolyl group, 2benzo oxazole group, 2-benzothiazolyl group, 2-benzo imidazolyl group, 4-methylphenyl group, 3methylphenyl group, 2-methylphenyl group, 2, 3-dimethylphenyl group, 2, 4-dimethylphenyl group, 2, 5-dimethylphenyl group, 2, 6-dimethylphenyl group, 3, 4-dimethylphenyl group, 3, 5-dimethylphenyl group, 2, 3, 4-trimethyl phenyl group, 2 and 3, 5-trimethyl phenyl group, 2, 3, 6-trimethyl phenyl group, 3 and 4, 5-trimethyl phenyl group, 4-ethyl phenyl group, 3-ethyl phenyl group, 2-ethyl phenyl group, a 2,3-diethyl phenyl group, a 2,4-diethyl phenyl group, a 2,5-diethyl phenyl group, a 2,6-diethyl phenyl group, a 3,4-diethyl phenyl group. A 3,5-diethyl phenyl group, 2 and 3, 4-triethyl phenyl group, 2, 3, 5triethyl phenyl group, 2 and 3, 6-triethyl phenyl group, A 3,4,5-triethyl phenyl group, a 4-n-propyl phenyl group, 4-isopropyl phenyl group, 2-isopropyl phenyl group, a 4-n-buthylphenyl group, Although 4-isobutyl phenyl group, a 4-sec-buthylphenyl group, a 4-tert-buthylphenyl group, a 3-tert-buthylphenyl group, a 2-tert-buthylphenyl group, etc. can be mentioned, it is not limited to these. [0039] As a bathophenanthroline compound of this invention, although the following compounds

[0039]As a bathophenanthroline compound of this invention, although the following compounds (illustration compounds 1-178) can be mentioned, for example, this invention is not limited to these (however, as for Me, a methyl group and Et express an ethyl group, Pr expresses a propyl group, and Bu expresses a butyl group.).

[0040]

[Formula 12]

[0044] [Formula 14]

[Formula 18]

例示化合物54

例示化合物58 [0049] [Formula 19]

例示化合物60

例示化合物59

[0051] [Formula 21]

例示化合物 67

例示化合物 68

例示化合物69



例示化合物70

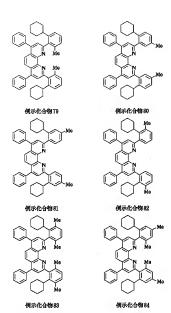


例示化合物71

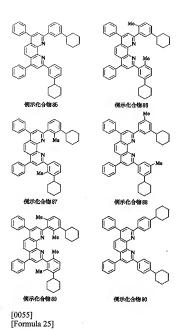
例示化合物72

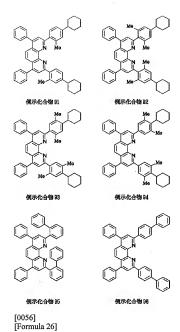
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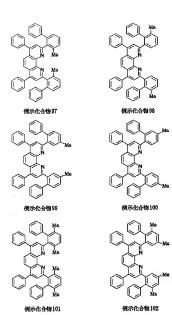
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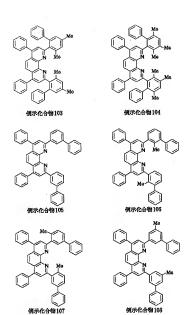
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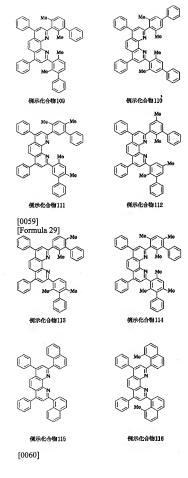




[0057] [Formula 27]



[0058] [Formula 28]



[Formula 30]





例示化合物117

例示化合物118

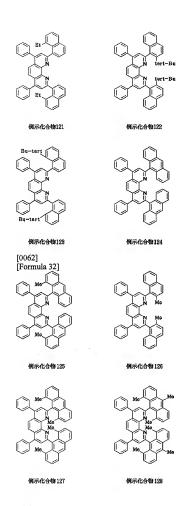


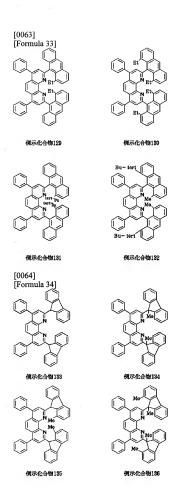


例示化合物119

例示化合物 120

[0061] [Formula 31]





[0065] [Formula 35]	CH ₀
例示化合物 187	例示化合物 188
CH Me	Mo Mo
例示化合物188	例示化合物 140
[0066] [Formula 36] ————————————————————————————————————	OR Mo
Mo CH2 Mo Mo Mo	Office Ma
例示化合物148	例示化合物144
[0067]	

例示化合物161

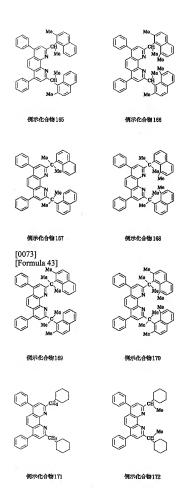
例示化合物162

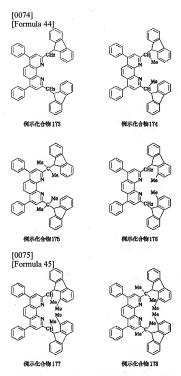


例示化合物183

例示化合物 184

[0072] [Formula 42]





[0076]Hereafter, the desirable embodiment which applied the bathophenanthroline compound of this invention to the organic EL device is described.

[0077]<1st embodiment> drawing 1 is an outline sectional view showing the important section of the organic EL device of the blue light nature by a 1st embodiment of this invention.

[0078]In this embodiment, the transparent electrode 5 which consists of an indium oxidation thing etc. which doped ITO (Indium Tin Oxide) and Zn is formed by methods, such as sputtering or vacuum deposition, on the glass substrate 6, The bathophenanthroline compound (derivative) contained layer 33 of the above-mentioned general formula which has the hole transporting bed 4a, the hole transportability luminous layer 4b, and hole block nature one by one on it, the electron transport layer 2, and the cathode

terminal 1 are laminated with a vacuum deposition method, The organic electroluminescence devices (organic EL device) 21 which consist of an amorphous organic thin film are produced. [0079]The hole transporting bed 4 is constituted as a structure which has the performance as a luminous layer, and this organic EL device 21 of other embodiments mentioned later is [that basic structure] the same.

[0080]Since the above-mentioned bathophenanthroline derivative contained layer 33 is inserted and laminated between the hole transporting bed 4 and the electron transport layer 2 as hole block layers, the feature of the organic EL device 21 of this embodiment, It is that promote recombination of an electronic-hole in inside of the hole transporting bed 4, and luminescence by the hole transporting bed 4 is obtained, and/or luminescence from the bathophenanthroline derivative contained layer 33 is also obtained.

[0081] <u>Drawing 2 shows typically a laminated structure of this above-mentioned embodiment (drawing 1) by a band scheme.</u>

[1082]A thick line (L_1, L_2) shown in a layer of the cathode 1 which consists of aluminum and aluminum-Li (aluminum lithium), and the ITO transparent electrode 5 in $\frac{1}{2}$ Are a near work function of each metal and in each class between these two electrodes Upside thick line $1_1, 1_2, 1_3, 1_4$, and a numerical value show a level of each minimum non-occupying molecular orbital (LUMO), and downward thick line $1_5, 1_6, 1_7, 1_8$, and numerical value show a level of each highest occupancy molecular orbital (HOMO). however, an energy level value in $\frac{1}{2}$ san example, is boiled variously and changes with construction material.

[0083]In this organic EL device, as shown in drawing 2, the hole h poured in from the transparent electrode 5 as an anode moves through the hole transporting bed 4, On the other hand, the electron e poured in from the metal electrode 1 of a cathode moves through the electron transport layer 2, this electronic-hole recombines in the hole transportability luminous layer 4, and luminescence is produced. [0084]The electron e poured in from the metal electrode 1 as a cathode. Since there is character which moves to the lower one of energy level, the metal electrode 1, the electron transport layer 2, the hole block layers 33, the hole transportability luminous layer 4b, The hole transportability luminous layers 4b and 4a can be reached in order of the hole transporting bed 4a via minimum non-occupying molecular orbital (LUMO) level 1, of each class - 14.

[0085]On the other hand, the hole h poured in from the ITO transparent electrode 5 as an anode. Since there is character which moves to the higher one of energy level, it can move to the electron transport layer 2 via highest occupancy molecular orbital (HOMO) level 15 of each class - 17 in order of the hole transporting bed 4a, the hole transportability luminous layer 4b, and the hole block layers 33. [0086] As shown in drawing 2, however, since the highest occupancy molecular orbital (HOMO) level lo of the electron transport layer 2 is lower in energy than in highest occupancy molecular orbital (HOMO) level 17 of the hole block layers 33, It becomes difficult to move to the electron transport layer 2 from the hole block layers 33, and the hole block layers 33 come to be filled with the poured-in hole h. [0087] As a result, the hole h with which the hole block layers 33 were filled promotes recombination of an electronic-hole in the hole transporting bed 4, and makes a luminescent material of the hole transportability luminous layers 4a and 4b which constitute the hole transporting bed 4 emit light. [0088] Thus, in the hole block layers 33, transportation of the hole h is effectively controlled by forming the hole block layers 33 to produce recombination of an electronic-hole efficiently in the hole transporting bed 4. And luminescence of the hole transportability luminous layer 4a is also added to luminescence by the hole transportability luminous layer 4b which mainly adjoins the hole block layers 33 among the hole transportability luminous layers 4a and 4b which emit light by this, and light of a specified wavelength (blue) is emitted.

[0089]Originally, in each layer, recombination of an electronic-hole produces the electron transport layer 2 and the hole transporting bed 4 by pouring of an electron from the cathode terminal 1, and pouring of a hole from the anode electrode 5. Therefore, when the above-mentioned ** hole block layers

33 do not exist, recombination of an electronic-hole arises in an interface of the electron transport layer 2 and the hole transporting bed 4, and only luminescence of long wavelength is obtained. However, it becomes possible like this example to promote blue light by making into a luminous region the hole transporting bed 4 which a luminescent substance contains by forming the hole block layers 33. [0090]As mentioned above, are for the hole block layers 33 controlling transportation of the hole h, and for the purpose, the highest occupancy molecular orbital (HOMO) level of a level with the lower highest occupancy molecular orbital (HOMO) level of a level with the lower highest occupancy molecular orbital (HOMO) level of a level with the lower highest occupancy molecular orbital (HOMO) level in energy of the hole transportability luminous layer 4b and the electron transport layer 2, and, Minimum a non-occupying molecular orbital of the hole block layers 33 (LUMO) A minimum non-occupying molecular orbital (LUMO) level of the hole transportability luminous layer 4b and the electron transport layer 2, It is more than a minimum non-occupying molecular orbital (LUMO) level of the lower one in energy, what is necessary is just below a minimum non-occupying molecular orbital (LUMO) level of the higher one in energy, and it is not limited to the above-mentioned composition.

[0091]However, this invention does not have the above-mentioned energy level in a mentioned range, and does not prevent itself from bathophenanthroline compound contained layer emitting light. Hole block layers may be the laminated structures which consist of two or more layers.

[0092]The above-mentioned hole block layers 33 can be formed with the above-mentioned bathophenanthroline derivative and/or other construction material, and they may also change the thickness in the range which can hold the function. Although the thickness is good to consider it as 1A - 1000 A (0.1 nm - 100 nm), if thickness is not much thin, hole block ability will be imperfect and a recombination area will straddle a hole transporting bed and an electron transport layer easily, and if not much thick, light may not be emitted from an increase in membrane resistance.

[0093]The above-mentioned organic EL device 21 is produced using the vacuum evaporator 11 like drawing 3. The support means 13 of a couple fixed under the arm 12 is formed in an inside of this device, among the fixing means 13 and 13 of these both sides, the clear glass board 6 is placed upside down, and a stage mechanism (graphic display abbreviation) which can set the mask 22 is established. And under the glass substrate 6 and the mask 22, the shutter 14 supported by the pivot 14a is arranged, and the various deposition sources 28 of a prescribed number are arranged in the lower part. Each deposition source is heated with a resistance heating method by the power supply 29. EB (electron beam) heating method etc. are used for this heating if needed.

[0094]In an above device, the mask 22 is an object for pixels and the shutter 14 is an object for deposition materials. And it is for rotating focusing on the pivot 14a and the shutter 14 intercepting a vapor stream of material according to sublimation temperature of a deposition material. [0095]Drawing 4 is a top view showing an example of the organic EL device 21 produced with the above-mentioned vacuum evaporator. Namely, by a thickness of about 100 nm with a vacuum evaporator which described above the ITO transparent electrode 5 whose sizes are 2 mm x 2 mm on the glass substrate 6 whose sizes L are 30 mm x 30 mm after vacuum evaporation, SiO_330 is vapor-

deposited on the whole surface, this is etched into a predetermined pixel pattern, many openings 31 are formed, and the transparent electrode 5 is exposed here, respectively. Therefore, each organic layers 4, 33, and 2 and the metal electrode 1 are formed one by one using the deposition mask 22 to luminous region (pixel) PX (2 mm x 2 mm) by SiO₂.

[0096]In this vacuum evaporator 11, a pixel with large size can also be independently formed in addition to what has the above-mentioned pixel of a large number like drawing 4.

[0097]As mentioned above, a stable organic EL device which has the hole transportability luminous layer 4 of a low voltage drive with high-intensity can be formed by laminating the organic layer 33 for improving efficiency of recombination of an electronic-hole all over a luminous region. About blue light, more than $10000\text{-cd}[/\text{m}\,]^2$ becomes possible [a pulse drive in 1 / 100 duty ratios] by direct-current drive to obtain luminosity more than peak luminance 2 of 55000cds/m by direct-current conversion so that it may mention later in detail especially.

- [0098]A transparent electrode, an organic hole transporting bed, organic hole block layers, an organic electron transport layer, and a metal electrode of the above-mentioned electroluminescence devices may be a laminated structure which each becomes from two or more layers.
- [0099]Each organic layer in the above-mentioned electroluminescence devices may be formed by methods, such as other methods for film deposition accompanied by sublimation or evaporation or a spin coat, and the cast, besides vacuum deposition.
- [0100]A hole transportability luminous layer of the above-mentioned electroluminescence devices may be an organic thin film which may perform a little vapor codeposition of a molecule for control of an emission spectrum of this element, for example, contains a little organic substances, such as a perylene derivative and a coumarin derivative.
- [0101]As a material usable as a hole carrying material, Benzidine or its derivative, styryl amine or its derivative, triphenylmethane, or its derivative is begun, Porphyrin or its derivative, triazole or its derivative, imidazole, or its derivative, Oxadiazole or its derivative, a poly aryl alkane, or its derivative, A phenylenediamine or its derivative, arylamine, or its derivative, Oxazol or its derivative, anthracene or its derivative, fluorenone, or its derivative, A monomer of heterocyclic conjugated systems, such as hydrazone or its derivative, a stilbene, its derivative or a polysilane system compound, a vinylearbazole system compound, a thiophene system compound, and an aniline system compound, oligomer, polymer, etc. are mentioned.
- [0102] Specifically alpha-naphthylphenyl diamine, porphyrin, metal tetraphenylporphyrin, Metal naphthalocyanine, a 4,4',4"-trimethyl triphenylamine, A 4,4',4"-tris(3-methylphenyl phenylamino) triphenylamine, N, N, N', N'-tetrakis (p-tolyl) p-phenylene diamine, Although N,N,N',N'-tetraphenyl-4,4'-diaminobiphenyl, N-phenylcarbazole, a 4-di-p-tolylamino stilbene, poly
- (PARAFENIREMBINIREN), poly (thiophene vinylene), poly (2,2'-thienyl pyrrole), etc. are mentioned, It is not limited to these.
- [0103]As a material usable as an electron-transport-property material, quinoline or its derivative, perylene or its derivative, screw styryl or its derivative, pyrazine, or its derivative is mentioned.
- [0104]Specifically, 8-hydroxy kino RINARU minium, anthracene, naphthalene, phenanthrene, pyrene, a chrysene, perylene, butadiene, a coumarin, an acridine, stilbenes, or these derivatives are mentioned. [0105]There is no restriction also in the material of construction, such as an anode electrode of the above-mentioned electroluminescence devices, and a cathode terminal.
- [0106]In order to pour in an electron efficiently about a cathode electrode material, it is preferred to use small metal of a work function from vacuum level of an electrode material, Besides an aluminium-lithium alloy, it is a simple substance about low work function metals, such as aluminum, indium, magnesium, silver, calcium, barium, and lithium, or may be used as an alloy with other metal, for example, improving stability.
- [0107]In order to take out organic electroluminescence from the anode electrode side, an example mentioned later used for an anode electrode ITO which is a transparent electrode, but. In order to pour in a hole efficiently, an electrode of a thing which has a large work function from vacuum level of an anode electrode material, for example, gold, a diacid-ized *****- antimony mixture, and a zinc oxide-aluminum mixture may be used.
- [0108]As the substrate 2, not only a glass substrate but opaque construction material, for example, a silicon substrate, Cr board, etc. may be used, and a substrate which formed metal by vacuum evaporation etc. or glass may be used. When opaque construction material is used for a substrate, in order to take out luminescence outside, it is preferred to be formed with construction material transparent [the upper surface (cathode terminal side) of an organic electroluminescence device] or translucent, and it can use ITO etc. for such materials.
- [0109]An object for full color which emits light in three colors of R, G, and B, or organic electroluminescence devices for multicolor are producible by choosing a luminescent material as well as organic electroluminescence devices for mono colors. In addition, this invention is applicable to organic electroluminescence devices usable also as an object for light sources not only as an object for a display, and it is applicable to other optical uses.

- [0110] The above-mentioned organic electroluminescence devices may drive an element in the state where close by a germanium oxidation thing etc. and eliminate influence of oxygen in the atmosphere, etc. in order to improve stability. and it lengthened to a vacuum.
- [0111]<2nd embodiment> drawing 5 is an outline sectional view showing an important section of an organic EL device by a 2nd embodiment of this invention.
- [0112]In the organic EL device 22 by this embodiment, forming the hole transportability luminous layer 4b, and forming a hole transportability luminous layer on the ITO transparent electrode 5, at a monolayer compared with an element of drawing 1, differ.
- [0113]<3rd embodiment> drawing 6 is an outline sectional view showing an important section of organic electroluminescence devices by a 3rd embodiment of this invention.
- [0114]In the organic EL device 23 by this embodiment, a hole transportability luminous layer is formed in a monolayer compared with an element of <u>drawing 1</u> like a 2nd embodiment that formed and described above the hole transporting bed (it serves as a hole transportability luminous layer) 4a on the TTO transparent electrode 5.

[Translation done.]

* NOTICES *

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- 1. This document has been translated by computer. So the translation may not reflect the original precisely.
- 2.**** shows the word which can not be translated.
- 3.In the drawings, any words are not translated.

EXAMPLE

[Example]Hereafter, this invention is explained still in detail about an example.

[0116]Composition [of Example 1 < 2 and 9-JI (2-methylphenyl) bathophenanthroline] > [0117] [Formula 46]

[0118] n-butyl lithium (a 1.6 M n-hexane solution, $17.0 \ ml, 26.8 mmol)$ was gradually dropped at the n-hexane solution (40 ml) of 2-iodotoluene (5.84 g, 26.4 mmol) under the room temperature. After

dropping, after stirring a reaction solution under a room temperature further for 16 hours, the ** exception carried out output and the white solid was washed by n-hexane (40mlx3). Under the room temperature, after the toluene solution (50 ml) of bathophenanthroline (2.03 g, 6.11mmol) was gradually dropped at anhydrous diethylether / toluene (3/1) solution of the obtained white solid (20 ml), in it, it was stirred under the room temperature for 16 hours.

[0119]60 ml of water ice-cooled to the obtained reaction solution was added, and the organic layer was separated. The organic layer obtained by chloroform extracting a water layer 3 times was mixed with the organic layer separated previously. After adding 60 g of manganese dioxide (chemical treatment article) to the mixed organic layer and stirring for 30 minutes, further, the sodium sulfate 100g was added and it stirred for 30 minutes.

[0120]The obtained mixed solution is filtered and condensed and column chromatography (silica gel, a developing solvent: n-hexane/chloroform =4/1->2/1) refines residue.

Then, the object compound (1.01 g, 49.5% of yield) was obtained as a light yellow crystal with recrystallization (recrystallization solvent: chloroform/n-hexane=2/1).

[0121] H-NMR (solvent; chloroform) and FAB-MS measurement performed immobilization of this output.

 1 H-NMR:2.70 (m, 6H, CH $_{3}$ -Ar-), 7.25-7.75 (s, 18H, aromatic), 7.80 (s, 2H, aromatic), and 7.90 (s, 2H, aromatic).

MS:m/s(relative intensity) 512 (M⁺, 100).

[0122] The visible absorption maximum wavelength of the tetrahydrofuran (THF) solution of this output was 297 nm, and the fluorescence wavelength was 390 nm.

[0123]Composition [of Example 2<2 and 9-JI (2, 6-dimethylphenyl) bathophenanthroline] > [0124] [Formula 47]

[0125]n-butyl lithium (a 1.6M n-hexane solution, 60.2 ml, 96.3mmol) was gradually dropped at the n-hexane / anhydrous diethylether (10/1) solution (110 ml) of 2-bromo-m-xylene (17.8 g, 96.3mmol) under the room temperature. After carrying out heating flowing back for 2 hours and stirring a reaction solution under a room temperature further after dropping for 16 hours, the ** exception carried out output and the white solid was washed by n-hexane (50mlx3). The toluene solution (80 ml) of bathophenanthroline (5.09 g, 15.3mmol) was gradually dropped at the anhydrous diethylether solution (40 ml) of the obtained white solid under the room temperature. After dropping, heating flowing back was carried out for 2 hours, and the reaction solution was stirred under the room temperature for 16 hours.

[0126]60 ml of water ice-cooled to the obtained reaction solution was added gradually, and the organic layer was separated. The organic layer obtained by chloroform extracting a water layer 3 times was

mixed with the organic layer separated previously. After adding 60 g of manganese dioxide (chemical treatment article) to the mixed organic layer and stirring for 30 minutes, further, the sodium sulfate 100g was added and it stirred for 30 minutes.

[0127]The obtained mixed solution is filtered and condensed and column chromatography (silica gel, a developing solvent: n-hexane/chloroform =8/1>>4/1) refines residue.

Then, the object compound (2.00 g, 39.4% of yield) was obtained as a light yellow crystal with recrystallization (recrystallization solvent: chloroform/n-hexane=2/1).

[0128] H-NMR and FAB-MS measurement performed immobilization of this output.

¹H-NMR:2.25 (m, 12H, CH₃-Ar-), 7.05-7.25 (s, 6H, aromatic), 7.35-7.70 (s, 12H, aromatic), and 7.95 (s, 2H, aromatic).

MS:m/s(relative intensity) 540 (M⁺, 100).

[0129]The visible absorption maximum wavelength of the THF solution of this output was 286 nm, and the fluorescence wavelength was 380 nm.

[0130]Composition [of Example 3<2 and 9-dinaphthyl bathophenanthroline] > [0131] [Formula 48]

[0132]n-butyl lithium (a 1.6M n-hexane solution, 15.3 ml, 24.4mmol) was gradually dropped at the n-hexane / anhydrous diethylether (1/1) solution (60 ml) of 1-bromonaphthalene (5.01 g, 24.4mmol) at 0 **. After dropping, under the room temperature, after stirring for 16 hours, the ** exception carried out output and residue was washed by n-hexane (40mlx3). The toluene solution (80 ml) of

bathophenanthroline (2.03 g, 6.1 lmmol) was gradually dropped at the anhydrous diethylether solution (40 ml) of the obtained solid under the room temperature. The reaction solution was stirred under the room temperature after dropping for 16 hours.

[0133]60 ml of water ice-cooled to the obtained reaction solution was added gradually, and the organic layer was separated. The organic layer obtained by chloroform extracting a water layer 3 times was mixed with the organic layer separated previously. After adding 60 g of manganese dioxide (chemical treatment article) to the mixed organic layer and stirring for 30 minutes, further, the sodium sulfate 100g was added and it stirred for 30 minutes.

[0134]The obtained mixed solution is filtered and condensed and column chromatography (silica gel, a developing solvent: n-hexane/chloroform =8/1->4/1) refines residue.

Then, the object compound (1.38 g, 68.2% of yield) was obtained with recrystallization (recrystallization solvent: chloroform/n-hexane=2/1).

[0135] H-NMR and FAB-MS measurement performed immobilization of this output.

H-NMR: 7.30-8.00 (s. 24H, aromatic), 8.32 (s. 2H, aromatic), and 8.68 (s. 2H, aromatic).

MS:m/s(relative intensity) 584 (M⁺, 100).

[0136]Composition [of Example 4< 2 and 9-JIFURUORENI roux bathophenanthroline] > [0137] [Formula 49]

[0138]Lithium diisopropylamine (LDA) (1.89 g, 17.4mmol) was added to the THF solution (30 ml) of the fluorene (4.16 g, 25.0mmol), and it stirred under the room temperature for 16 hours. Then, decompression distilling off of THF and the diisopropylamine was carried out. The toluene solution (60 ml) of bathophenanthroline (2.03 g, 6.11mmol) was gradually dropped at the anhydrous diethylether solution (20 ml) of the obtained yellow solid under the room temperature. After dropping, heating flowing back was carried out for 2 hours, and the reaction solution was stirred under the room temperature for 16 hours.

[0139]60 ml of water ice-cooled to the obtained reaction solution was added gradually, and the organic layer was separated. The organic layer obtained by chloroform extracting a water layer 3 times was mixed with the organic layer separated previously. After adding 60 g of manganese dioxide (chemical treatment article) to the mixed organic layer and stirring for 30 minutes, further, the sodium sulfate 100g was added and it stirred for 30 minutes.

[0140]The obtained mixed solution is filtered and condensed and column chromatography (silica gel, a developing solvent: n-hexane/chloroform =8/1->4/1) refines residue.

Then, the object compound (1.38 g, 68.2% of yield) was obtained with recrystallization (recrystallization solvent: chloroform/n-hexane=2/1).

[0141] H-NMR and FAB-MS measurement performed identification of this output. H-NMR:4.51 (m, 2H, Ar-CH₂-Ar), 7.30-7.78 (s, 28H, aromatic), and 7.81 (s, 2H, aromatic).

MS:m/s(relative intensity) 660 (M+, 100).

[0142]Example 5 < composition of 2,9-dibenzyl-bathophenanthroline> [0143]

[Formula 50]

[0144]n-butyl lithium (a 1.6M n-hexane solution, 4.45 ml, 7.13mmol) was gradually dropped at anhydrous toluene (2.24 g, 24.9mmol) under the room temperature. After dropping and after adding THF (1.06 g, 14.7mmol) over 30 minutes [Me-THF / (0.627 g, 7.47mmol) / for 20 minutes] at -22 ** after that, it stirred at 6-10 ** for 16 hours. The obtained reaction solution and the toluene solution (40 ml) of bathophenanthroline (2.03 g, 6.11mmol) were gradually dropped under the room temperature. The reaction solution was stirred under the room temperature after dropping for 16 hours.

[0145]60 ml of water ice-cooled to the obtained reaction solution was added gradually, and the organic layer was separated. The organic layer obtained by chloroform extracting a water layer 3 times was mixed with the organic layer separated previously. After adding 60 g of manganese dioxide (chemical treatment article) to the mixed organic layer and stirring for 30 minutes, further, the sodium sulfate 100g was added and it stirred for 30 minutes.

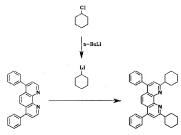
[0146]The obtained mixed solution is filtered and condensed and column chromatography (silica gel, a developing solvent: n-hexane/chloroform =8/1->4/1) refines residue.

Then, the object compound (0.88 g, 43.3% of yield) was obtained with recrystallization (recrystallization solvent: chloroform/n-hexane=2/1).

[0147] H-NMR and FAB-MS measurement performed identification of this output. H-NMR:4.68 (m, 4H, -CH₂-Ar), 7.28-7.78 (s, 22H, aromatic), and 7.81 (s, 2H, aromatic).

¹H-NMR:4.68 (m, 4H, -CH₂-Ar), 7.28-7.78 (s, 22H, aromatic), and 7.81 (s, 2H, aromatic MS:m/s(relative intensity) 512 (M⁺, 100).

[0148]Composition [of Example 6<2 and 9-dicyclohexyl bathophenanthroline] > [0149] [Formula 51]



[0150]n-butyl lithium (a 1.6M n-hexane solution, 36.3 ml, 58.0mmol) was gradually dropped at chlorocyclohexane (3.00 g, 25.0mmol) under the room temperature at n-hexane / anhydrous diethylether (10/1) solution (50 ml). After dropping, after stirring a reaction solution under a room temperature further for 16 hours, the ** exception carried out output and the white solid was washed by n-hexane (50mk3). The toluene solution (40 ml) of bathophenanthroline (2.03 g, 6.11mmol) was gradually dropped at the anhydrous diethylether solution (10 ml) of the obtained white solid under the room temperature. The reaction solution was stirred under the room temperature after dropping for 16 hours. [0151]60 ml of water ice-cooled to the obtained reaction solution was added gradually, and the organic layer was separated. The organic layer obtained by chloroform extracting a water layer 3 times was mixed with the organic layer separated previously. After adding 60 g of manganese dioxide (chemical treatment article) to the mixed organic layer and stirring for 30 minutes, further, the sodium sulfate 100g was added and it stirred for 30 minutes.

[0152]The obtained mixed solution is filtered and condensed and column chromatography (silica gel, a developing solvent: n-hexane/chloroform =8/1->4/1) refines residue.

Then, the object compound (0.98 g, 48.3% of yield) was obtained with recrystallization (recrystallization solvent: chloroform/n-hexane=2/1).

[0153] H-NMR and FAB-MS measurement performed identification of this output.

¹H-NMR:0.80-2.45 (m, 20H, -CH₂-CH₂-CH₂-CH₂-CH₂-), 3.20 (m, 2H, -CH-Ar), 7.25-7.75 (s, 12H, aromatic), 7.81 (s, 2H, aromatic).

MS:m/s(relative intensity) 496 (M⁺, 100).

[0154]Composition [of Example 7 < 2 and 9-JIBIFENI roux bathophenanthroline] > [0155] [Formula 52]

[0156]n-butyl lithium (a 1.6M n-hexane solution, 17.0 ml, 27.2mmol) was gradually dropped at 4-bromobiphenyl (6.33 g, 27.2mmol) under the room temperature at n-hexane / anhydrous diethylether (10/1) solution (110 ml). After dropping, after stirring a reaction solution under a room temperature for 16 hours, the ** exception carried out output and the white solid was washed by n-hexane (50mlx3). The toluene solution (40 ml) of bathophenanthroline (2.03 g, 6.11mmol) was gradually dropped at the anhydrous diethylether solution (20 ml) of the obtained white solid under the room temperature. The reaction solution was stirred under the room temperature after dropping for 16 hours.

[0157]60 ml of water ice-cooled to the obtained reaction solution was added gradually, and the organic layer was separated. The organic layer obtained by chloroform extracting a water layer 3 times was mixed with the organic layer separated previously. After adding 60 g of manganese dioxide (chemical treatment article) to the mixed organic layer and stirring for 30 minutes, further, the sodium sulfate 100g was added and it stirred for 30 minutes.

[0158]The obtained mixed solution is filtered and condensed and column chromatography (silica gel, a developing solvent: n-hexane/chloroform =8/1->4/1) refines residue.

Then, the object compound (0.76 g, 37.4% of yield) was obtained with recrystallization (recrystallization solvent: chloroform/n-hexane=2/1).

[0159] H-NMR and FAB-MS measurement performed identification of this output.

H-NMR: 7.25-7.78 (s. 26H, aromatic), 7.81 (s. 2H, aromatic), and 8.32 (s. 4H, aromatic).

MS:m/s(relative intensity) 636 (M⁺, 100).

[0160]Example 8 < composition of 2,9-JI (2-methylbenzyl)-bathophenanthroline> [0161] [Formula 53]

[0162]n-butyl lithium (a 1.6M n-hexane solution, 4.45 ml, 7.13mmol) was gradually dropped at alphabromo-o-xylene (4.91 g, 24.9mmol) under the room temperature. After dropping and after adding THF (1.06 g, 14.7mmol) ver 30 minutes [Me-THF / (0.627 g, 7.47mmol) / for 20 minutes] at -22 ** after that, it stirred at 6-10 ** for 16 hours. The obtained reaction solution and the toluene solution (40 ml) of bathophenanthroline (2.03 g, 6.11mmol) were gradually dropped under the room temperature. The reaction solution was stirred under the room temperature after dropping for 16 hours.

[0163]60 ml of water ice-cooled to the obtained reaction solution was added gradually, and the organic layer was separated. The organic layer obtained by chloroform extracting a water layer 3 times was mixed with the organic layer separated previously. After adding 60 g of manganese dioxide (chemical reatment article) to the mixed organic layer and stirring for 30 minutes, further, the sodium sulfate 100g was added and it stirred for 30 minutes.

[0164]The obtained mixed solution is filtered and condensed and column chromatography (silica gel, a developing solvent: n-hexane/chloroform =8/1->4/1) refines residue.

Then, the object compound (0.72 g, 35.4% of yield) was obtained with recrystallization (recrystallization solvent: chloroform/n-hexane=2/1).

[0165] H-NMR and FAB-MS measurement performed identification of this output.

¹H-NMR:2.35 (m, 6H, CH₃-Ar-), 4.65 (m, 4H, CH₂-Ar-), 7.25-7.78 (s, 20H, aromatic), and 7.81 (s, 2H, aromatic).

MS:m/s(relative intensity) 540 (M+, 100).

[0166] Example 9 < composition of 2,9-JÍ (8-methylnaphthyl)-bathophenanthroline> [0167] [Formula 54]

[0168]n-butyl lithium (a 1.6M n-hexane solution, 15.3 ml, 24.4mmol) was gradually dropped at 1bromo-8-methylnaphthalene (5.34 g, 24.4mmol) at 0 ** at n-hexane / anhydrous diethylether (1/1) solution (60 ml). After dropping, under the room temperature, after stirring for 16 hours, the ** exception carried out output and residue was washed by n-hexane (40mlx3). The toluene solution (80 ml) of bathophenanthroline (2.03 g, 6.11mmol) was gradually dropped at the anhydrous diethylether solution (40 ml) of the obtained solid under the room temperature. The reaction solution was stirred under the room temperature after dropping for 16 hours.

[0169]60 ml of water ice-cooled to the obtained reaction solution was added gradually, and the organic layer was separated. The organic layer obtained by chloroform extracting a water layer 3 times was mixed with the organic layer separated previously. After adding 60 g of manganese dioxide (chemical treatment article) to the mixed organic layer and stirring for 30 minutes, further, the sodium sulfate 100g was added and it stirred for 30 minutes.

[0170] The obtained mixed solution is filtered and condensed and column chromatography (silica gel, a developing solvent: n-hexane/chloroform =8/1->4/1) refines residue.

Then, the object compound (1.30 g, 64.0% of yield) was obtained with recrystallization (recrystallization solvent: chloroform/n-hexane=2/1).

[0171] H-NMR and FAB-MS measurement performed identification of this output.

¹H-NMR:2.60 (m, 6H, CH₃-Ar-), 7.30-7.81 (s, 22H, aromatic), 7.81 (s, 2H, aromatic), and 8.25 (s, 2H, aromatic).

MS:m/s(relative intensity) 612 (M⁺, 100).

[0172] Example 10 < composition of 2,9-JI (2-methylnaphthyl)-bathophenanthroline> [0173]

[Formula 55]

[0174]n-butyl lithium (a 1.6M n-hexane solution, 15.3 ml, 24.4mmol) was gradually dropped at the n-hexane / anhydrous diethylether (1/1) solution (60 ml) of 1-bromo-2-methylnaphthalene (5.34 g, 24.4mmol) at 0 **. After dropping, under the room temperature, after stirring for 16 hours, the ** exception carried out output and residue was washed by n-hexane (40mlx3). The toluene solution (80 ml) of bathophenanthroline (2.03 g, 6.11mmol) was gradually dropped at the anhydrous diethylether solution (40 ml) of the obtained solid under the room temperature. The reaction solution was stirred under the room temperature after dropping for 16 hours.

[0175]60 ml of water ice-cooled to the obtained reaction solution was added gradually, and the organic layer was separated. The organic layer obtained by chloroform extracting a water layer 3 times was mixed with the organic layer separated previously. After adding 60 g of manganese dioxide (chemical treatment article) to the mixed organic layer and stirring for 30 minutes, further, the sodium sulfate 100g was added and it stirred for 30 minutes.

[0176]The obtained mixed solution is filtered and condensed and column chromatography (silica gel, a developing solvent: n-hexane/chloroform =8/1->4/1) refines residue.

Then, the object compound (1.20 g, 59.1% of yield) was obtained with recrystallization (recrystallization solvent: chloroform/n-hexane=2/1).

 $[0177]^{1}$ H-NMR and FAB-MS measurement performed identification of this output. 1 H-NMR:2.80 (m, 6H, CH₃-Ar-), 7.25-7.78 (s, 24H, aromatic), and 7.81 (s, 2H, aromatic).

MS:m/s(relative intensity) 612 (M+, 100).

[0178] Example 11 <composition of 2,9-JI (alpha-methylbenzyl)-bathophenanthroline> [0179] [Formula 56]

[0180]n-butyl lithium (a 1.6M n-hexane solution, 4.45 ml, 7.13mmol) was gradually dropped at 1-bromo-1-phenylethane (4.91 g, 24.9mmol) under the room temperature. After dropping and after adding THF (1.06 g, 14.7mmol) over 30 minutes [Me-THF / (0.627 g, 7.47mmol) / for 20 minutes] at -22 ** after that, it stirred at 6-10 ** for 16 hours. The obtained reaction solution and the toluene solution (40 ml) of bathophenanthroline (2.03 g, 6.11mmol) were gradually dropped under the room temperature. The reaction solution was stirred under the room temperature after dropping for 16 hours. [0181]60 ml of water ice-cooled to the obtained reaction solution was added gradually, and the organic layer was separated. The organic layer obtained by chloroform extracting a water layer 3 times was mixed with the organic layer separated previously. After adding 60 g of manganese dioxide (chemical treatment article) to the mixed organic layer and stirring for 30 minutes, further, the sodium sulfate 100g was added and it stirred for 30 minutes.

[0182]The obtained mixed solution is filtered and condensed and column chromatography (silica gel, a developing solvent: n-hexane/chloroform=8/1>4/1) refines residue. Then, the object compound (0.83 g, 40.9% of yield) was obtained with recrystallization

Then, the object compound (0.83 g, 40.9% of yield) was obtained with recrystallizatio (recrystallization solvent: chloroform/n-hexane=2/1).

[0183] H-NMR and FAB-MS measurement performed identification of this output.

¹H-NMR:2.40 (m, 6H, CH₃-Ar-), 4.64 (m, 2H, -CH-Ar), 7.25-7.78 (s, 22H, aromatic), and 7.81 (s, 2H, aromatic).

MS:m/s(relative intensity) 540 (M⁺, 100).

[Translation done.]

DESCRIPTION OF DRAWINGS

[Brief Description of the Drawings]

 $[\underline{Drawing\ 1}] It is an outline sectional view of the important section of the organic EL device using the bathophenanthroline compound of this invention.$

[<u>Drawing 2</u>]It is a band scheme figure showing the laminated structure of a **** organic EL device typically.

[Drawing 3]It is an outline sectional view of the vacuum evaporator used for manufacture of a ****
organic EL device.

[Drawing 4]It is a top view of a **** organic EL device.

[Drawing 5]It is an outline sectional view of the important section of other organic EL devices using the bathophenanthroline compound of this invention.

[<u>Drawing 6</u>]Furthermore it used the bathophenanthroline compound of this invention, it is an outline sectional view of the important section of other organic EL devices.

[Drawing 7]It is an outline sectional view showing an example of the conventional organic EL device. [Drawing 8]It is an outline sectional view showing an example of an organic EL device besides ****. [Drawing 9]It is an outline perspective view showing the example of a **** organic EL device.

[Description of Notations]

Î [-- A hole transportability luminous layer, 5 / -- An ITO transparent electrode (anode), 6 / -- A glass substrate, 10, 21 22, 23 / -- An organic EL device, 33 / -- Hole block layers, e / -- An electron, h / -- Hole | -- A metal electrode (cathode), 2 -- An electron transport layer, 4 -- A hole transporting bed, 4a, 4b

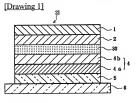
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* NOTICES *

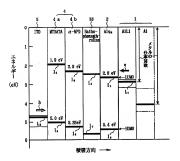
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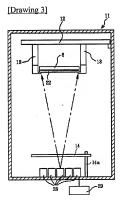
- 1. This document has been translated by computer. So the translation may not reflect the original precisely.
- 2.**** shows the word which can not be translated.
- In the drawings, any words are not translated.

DRAWINGS

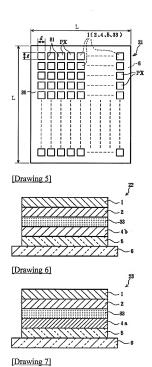


[Drawing 2]

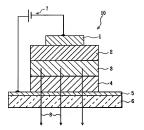


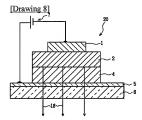


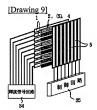
[Drawing 4]



3/6/2009







[Translation done.]

CORRECTION OR AMENDMENT

[Kind of official gazette]Printing of amendment by the regulation of 2 of Article 17 of Patent Law [Section classification] The 2nd classification of the part III gate [Publication date]December 24, Heisei 16 (2004.12.24)

[Publication No.]JP,2001-131174,A (P2001-131174A)

[Date of Publication]May 15, Heisei 13 (2001.5.15) [Application number]Japanese Patent Application No. 11-312071 [The 7th edition of International Patent Classification]

C07D471/04

H05B 33/14

H05B 33/22

[FI]

C07D471/04 112 T

H05B 33/14 B

H05B 33/22 B

[Written amendment]

[Filing date]January 26, Heisei 16 (2004.1.26)

[Amendment 1]

[Document to be Amended]Specification

[Item(s) to be Amended]Claim 3

[Method of Amendment]Change [The contents of amendment]

[Claim 3]

A bathophenanthroline compound indicated to claim 1 or 2. [available as organic materials used for organic electroluminescence devices, for example, a carrier transport material]

[Translation done.]